

December 06, 2016

Tom Moe  
USS Corporation  
P.O. Box 417  
8771 Park Ridge Dr  
Mountain Iron, MN 55768

RE: Project: Toxicity  
Pace Project No.: 1279287

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on November 21, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,



Melisa M Woods for  
Dan J Toms  
dan.toms@pacelabs.com  
Project Manager

Enclosures



## REPORT OF LABORATORY ANALYSIS

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## CERTIFICATIONS

Project: Toxicity  
Pace Project No.: 1279287

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### **Duluth Minnesota Cerification ID's**

4730 Oneota St., Duluth, MN 55807

Minnesota Dept of Health Certification #: 027-137-152

Wisconsin DNR Certification # : 999446800

North Dakota Certification #: R-105

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## REPORT OF LABORATORY ANALYSIS

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## SAMPLE SUMMARY

Project: Toxicity  
Pace Project No.: 1279287

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1279287001	WS006/WS007	Water	11/21/16 10:25	11/21/16 16:15
1279287002	SW002	Water	11/21/16 09:00	11/21/16 16:15

## REPORT OF LABORATORY ANALYSIS

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## SAMPLE ANALYTE COUNT

Project: Toxicity  
Pace Project No.: 1279287

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1279287001	WS006/WS007	EPA 120.1 (1982)	AXP	1	PASI-DUL
		SM 2320B (1997)	KJD	1	PASI-DUL
		SM 4500-CL E	DJT	1	PASI-DUL
		SM 4500-H+B (1996)	CJA	1	PASI-DUL
		SM 4500-NH3 D (1997)	AXP	1	PASI-DUL
		USGS I-1338-85 (1985)	KJD	1	PASI-DUL
1279287002	SW002	EPA 120.1 (1982)	AXP	1	PASI-DUL
		SM 2320B (1997)	KJD	1	PASI-DUL
		SM 4500-CL E	DJT	1	PASI-DUL
		SM 4500-H+B (1996)	CJA	1	PASI-DUL
		SM 4500-NH3 D (1997)	AXP	1	PASI-DUL
		USGS I-1338-85 (1985)	KJD	1	PASI-DUL

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## ANALYTICAL RESULTS

Project: Toxicity  
Pace Project No.: 1279287

Sample: WS006/WS007	Lab ID: 1279287001		Collected: 11/21/16 10:25		Received: 11/21/16 16:15		Matrix: Water	
Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
120.1 Specific Conductance	Analytical Method: EPA 120.1 (1982)							
Specific Conductance	2130	umhos/cm	1.0	1		11/30/16 14:29		
2320B Alkalinity	Analytical Method: SM 2320B (1997)							
Alkalinity, Total as CaCO3	259	mg/L	20.0	1		11/23/16 11:50		
4500CL E Chlorine, Residual	Analytical Method: SM 4500-CL E							
Chlorine, Total Residual	ND	mg/L	0.020	1		11/21/16 17:12	7782-50-5	H6
4500H+ pH, Electrometric	Analytical Method: SM 4500-H+B (1996)							
pH at 25 Degrees C	8.2	Std. Units	0.10	1		11/21/16 17:24		H6
4500 Ammonia Water	Analytical Method: SM 4500-NH3 D (1997)							
Nitrogen, Ammonia	1.2	mg/L	0.20	1		11/30/16 10:17	7664-41-7	
USGS Hardness, Total as CaCO3	Analytical Method: USGS I-1338-85 (1985)							
Total Hardness	1100	mg/L	5.0	1		11/23/16 14:57		

Sample: SW002	Lab ID: 1279287002		Collected: 11/21/16 09:00		Received: 11/21/16 16:15		Matrix: Water	
Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
120.1 Specific Conductance	Analytical Method: EPA 120.1 (1982)							
Specific Conductance	65.5	umhos/cm	1.0	1		11/30/16 14:29		
2320B Alkalinity	Analytical Method: SM 2320B (1997)							
Alkalinity, Total as CaCO3	20.7	mg/L	20.0	1		11/23/16 11:59		
4500CL E Chlorine, Residual	Analytical Method: SM 4500-CL E							
Chlorine, Total Residual	ND	mg/L	0.020	1		11/21/16 17:20	7782-50-5	H6
4500H+ pH, Electrometric	Analytical Method: SM 4500-H+B (1996)							
pH at 25 Degrees C	7.0	Std. Units	0.10	1		11/21/16 17:27		H6
4500 Ammonia Water	Analytical Method: SM 4500-NH3 D (1997)							
Nitrogen, Ammonia	ND	mg/L	0.20	1		11/30/16 10:24	7664-41-7	
USGS Hardness, Total as CaCO3	Analytical Method: USGS I-1338-85 (1985)							
Total Hardness	28.0	mg/L	5.0	1		11/23/16 15:05		

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## QUALITY CONTROL DATA

Project: Toxicity  
Pace Project No.: 1279287

QC Batch: 101087 Analysis Method: EPA 120.1 (1982)  
QC Batch Method: EPA 120.1 (1982) Analysis Description: 120.1 Specific Conductance  
Associated Lab Samples: 1279287001, 1279287002

METHOD BLANK: 401900 Matrix: Water  
Associated Lab Samples: 1279287001, 1279287002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Specific Conductance	umhos/cm	ND	1.0	11/30/16 14:24	

LABORATORY CONTROL SAMPLE: 401899

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Specific Conductance	umhos/cm	1410	1360	96	90-110	

SAMPLE DUPLICATE: 401901

Parameter	Units	1279472001 Result	Dup Result	RPD	Max RPD	Qualifiers
Specific Conductance	umhos/cm	122	122	0	10	

SAMPLE DUPLICATE: 401902

Parameter	Units	1279512002 Result	Dup Result	RPD	Max RPD	Qualifiers
Specific Conductance	umhos/cm	431	431	0	10	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: Toxicity  
Pace Project No.: 1279287

QC Batch: 100720 Analysis Method: SM 2320B (1997)  
QC Batch Method: SM 2320B (1997) Analysis Description: 2320B Alkalinity  
Associated Lab Samples: 1279287001, 1279287002

METHOD BLANK: 400340 Matrix: Water  
Associated Lab Samples: 1279287001, 1279287002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Alkalinity, Total as CaCO <sub>3</sub>	mg/L	ND	20.0	11/23/16 11:15	

LABORATORY CONTROL SAMPLE: 400339

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Alkalinity, Total as CaCO <sub>3</sub>	mg/L	50	48.6	97	90-110	

SAMPLE DUPLICATE: 400341

Parameter	Units	1279213001 Result	Dup Result	RPD	Max RPD	Qualifiers
Alkalinity, Total as CaCO <sub>3</sub>	mg/L	134	136	2	10	

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## QUALITY CONTROL DATA

Project: Toxicity  
Pace Project No.: 1279287

QC Batch:	100613	Analysis Method:	SM 4500-CL E
QC Batch Method:	SM 4500-CL E	Analysis Description:	4500CL E Chlorine, Total Residual
Associated Lab Samples:	1279287001, 1279287002		

METHOD BLANK: 399715      Matrix: Water  
Associated Lab Samples: 1279287001, 1279287002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Chlorine, Total Residual	mg/L	ND	0.020	11/21/16 17:10	H6

LABORATORY CONTROL SAMPLE: 399714

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Chlorine, Total Residual	mg/L	.1	0.097	97	80-120	H6

SAMPLE DUPLICATE: 399716

Parameter	Units	1279287001 Result	Dup Result	RPD	Max RPD	Qualifiers
Chlorine, Total Residual	mg/L	ND	ND		20	H6

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## QUALITY CONTROL DATA

Project: Toxicity  
Pace Project No.: 1279287

QC Batch: 100776 Analysis Method: SM 4500-H+B (1996)  
QC Batch Method: SM 4500-H+B (1996) Analysis Description: 4500H+B pH Electrometric  
Associated Lab Samples: 1279287001, 1279287002

LABORATORY CONTROL SAMPLE: 400592

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
pH at 25 Degrees C	Std. Units	7	7.0	101	98-102	H6

SAMPLE DUPLICATE: 400593

Parameter	Units	1279287001 Result	Dup Result	RPD	Max RPD	Qualifiers
pH at 25 Degrees C	Std. Units	8.2	8.2	0	10	H6

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## QUALITY CONTROL DATA

Project: Toxicity  
Pace Project No.: 1279287

QC Batch: 101025 Analysis Method: SM 4500-NH3 D (1997)  
QC Batch Method: SM 4500-NH3 D (1997) Analysis Description: 4500 Ammonia  
Associated Lab Samples: 1279287001, 1279287002

METHOD BLANK: 401601 Matrix: Water  
Associated Lab Samples: 1279287001, 1279287002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Nitrogen, Ammonia	mg/L	ND	0.20	11/30/16 09:53	

LABORATORY CONTROL SAMPLE: 401600

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Nitrogen, Ammonia	mg/L	2	1.9	94	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 401602 401603

Parameter	Units	1279472001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Nitrogen, Ammonia	mg/L	ND	2	2	1.9	2.0	95	98	90-110	4	10	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 401604 401605

Parameter	Units	1279511001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Nitrogen, Ammonia	mg/L	ND	2	2	2.0	2.1	98	101	90-110	2	10	

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## QUALITY CONTROL DATA

Project: Toxicity  
Pace Project No.: 1279287

QC Batch: 100778 Analysis Method: USGS I-1338-85 (1985)  
QC Batch Method: USGS I-1338-85 (1985) Analysis Description: USGS T Hardness as CaCO<sub>3</sub>  
Associated Lab Samples: 1279287001, 1279287002

METHOD BLANK: 400595 Matrix: Water  
Associated Lab Samples: 1279287001, 1279287002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Total Hardness	mg/L	ND	5.0	11/23/16 14:26	

LABORATORY CONTROL SAMPLE: 400596

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Hardness	mg/L	100	101	101	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 400597 400598

Parameter	Units	1279213001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Total Hardness	mg/L	320	200	200	528	526	104	103	90-110	0	10	

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## QUALIFIERS

Project: Toxicity  
Pace Project No.: 1279287

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### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### LABORATORIES

PASI-DUL Pace Analytical Services - Duluth

### ANALYTE QUALIFIERS

H6 Analysis initiated outside of the 15 minute EPA required holding time.

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## QUALITY CONTROL DATA CROSS REFERENCE TABLE


Project: Toxicity  
Pace Project No.: 1279287

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1279287001	WS006/WS007	EPA 120.1 (1982)	101087		
1279287002	SW002	EPA 120.1 (1982)	101087		
1279287001	WS006/WS007	SM 2320B (1997)	100720		
1279287002	SW002	SM 2320B (1997)	100720		
1279287001	WS006/WS007	SM 4500-CL E	100613		
1279287002	SW002	SM 4500-CL E	100613		
1279287001	WS006/WS007	SM 4500-H+B (1996)	100776		
1279287002	SW002	SM 4500-H+B (1996)	100776		
1279287001	WS006/WS007	SM 4500-NH3 D (1997)	101025		
1279287002	SW002	SM 4500-NH3 D (1997)	101025		
1279287001	WS006/WS007	USGS I-1338-85 (1985)	100778		
1279287002	SW002	USGS I-1338-85 (1985)	100778		

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	Document Name:	Document Revised: 22Jan2016
	Sample Condition Upon Receipt Form	Page 1 of 1
	Document No.: F-DUL-C-001-Rev.01	Issuing Authority: Pace Virginia, Minnesota Quality Office

**Sample Condition Upon Receipt**

Client Name:

Project #:

US Steel

WO#: **1279287**



Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☒ Client  
☐ Commercial ☐ Pace ☐ Other: \_\_\_\_\_

Tracking Number: \_\_\_\_\_

Custody Seal on Cooler/Box Present? ☐ Yes ☒ No Seals Intact? ☐ Yes ☒ No

Optional: Proj. Due Date: \_\_\_\_\_ Proj. Name: \_\_\_\_\_

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☒ None ☐ Other: \_\_\_\_\_ Temp Blank? ☐ Yes ☒ No

Thermometer Used: ☒ B00051 ☐ SW001 ☐ SW002 Type of Ice: ☐ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 2.0/1.0 Cooler Temp Corrected °C: 1.4/0.4 Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA  
Temp should be above freezing to 6°C Correction Factor: -0.6°C Date and Initials of Person Examining Contents: 11/21/16 Kp

Comments:

Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.
Sampler Name and Signature on COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.
Short Hold Time Analysis (<72 hr)?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	6. <u>pH, res cl</u>
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.
-Includes Date/Time/ID/Analysis Matrix: <u>WT</u>		
All containers needing acid/base preservation will be checked and documented in the pH logbook.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13.
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	14.
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
Pace Trip Blank Lot # (if purchased):		

**CLIENT NOTIFICATION/RESOLUTION**

Field Data Required? ☐ Yes ☐ No

Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

FECAL WAIVER ON FILE Y N

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review:

AP for DJT

Date:

11-21-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e. out of hold, incorrect preservative, out of temp, incorrect containers)